

## **THE CLAIMS**

### **What is claimed is:**

1. Large area single crystal III-V nitride material with uniformly low dislocation density on at least one surface thereof.
2. Material according to claim 1, selected from the group consisting of AlN, InN, GaN, AlInN, AlInGaN, InGaN, and AlGaN.
3. GaN material according to claim 1.
4. Material according to claim 1, doped with a dopant species.
5. Material according to claim 4, of a p-doped, n-doped or semi-insulatively doped character.
6. Material according to claim 3, doped with a dopant species.
7. Material according to claim 6, wherein the dopant species includes a dopant selected from the group consisting of oxygen and silicon.
8. Material according to claim 3, having a large area of greater than 2 cm<sup>2</sup>.
9. Material according to claim 3, having a large area of greater than 15 cm<sup>2</sup>.
10. Material according to claim 3, having a thickness of at least 0.1 mm.

11. Material according to claim 3, having an ADD not exceeding  $2 \times 10^6 \text{ cm}^{-2}$ .
12. Material according to claim 3, having an ADD not exceeding  $1 \times 10^6 \text{ cm}^{-2}$ .
13. Material according to claim 3, having an ADD not exceeding  $5 \times 10^5 \text{ cm}^{-2}$ .
14. Material according to claim 3, having a DDSDR of less than 50%.
15. Material according to claim 3, having a DDSDR of less than 25%.
16. Large area, uniformly low dislocation density single crystal gallium nitride, having a large area of greater than  $15 \text{ cm}^2$ , a thickness of at least 0.1 mm, an ADD not exceeding  $1 \times 10^6 \text{ cm}^{-2}$ , and a DDSDR of less than 25%.
17. An article, comprising material as claimed in claim 1.
18. The article of claim 17, wherein said material is on a heteroepitaxial substrate.
19. The article of claim 18, wherein the heteroepitaxial substrate comprises a material selected from the group consisting of sapphire, silicon carbide, gallium arsenide, silicon, lithium gallate, lithium aluminate, lithium aluminum gallate, zinc oxide, diamond, spinel, and magnesium oxide.
20. The article of claim 17, wherein said material is GaN.
21. The article of claim 20, in the form of a crystal having a thickness of at least  $50 \text{ }\mu\text{m}$ .
22. The article of claim 21, wherein the thickness of the crystal is greater than  $500 \text{ }\mu\text{m}$ .

23. The article of claim 21, wherein the thickness of the crystal is greater than 2 mm.
24. The article of claim 21, wherein the thickness of the crystal is greater than 10 mm.
25. The article of claim 21, having a surface including said large area, wherein said surface is at least 2 inches in diameter.
26. A wafer comprising large area single crystal gallium nitride material with uniformly low dislocation density on at least one surface thereof.
27. The wafer of claim 26, having a diameter of from 2 to 8 inches.
28. The wafer of claim 26, having a rectangular or square shape, with each side at least 15 mm in size.
29. The wafer of claim 26, having a surface that is parallel to the c-plane of the crystal plane of the single crystal gallium nitride material.
30. The wafer of claim 26, having a surface at an angle with the c-plane of the single crystal gallium nitride material.
31. The wafer of claim 30, wherein said angle is in a range of from about 0.1 to about 10 degrees.
32. The wafer of claim 26, having surfaces thereof polished to a mirror finish.
33. The wafer of claim 26, including a chemical mechanically polished gallium-terminated surface.

34. The wafer of claim 26, wherein said at least one surface includes a c-plane surface.
35. The wafer of claim 26, wherein said at least one surface includes a surface off-cut at an angle in a range of from about 0.2 to about 8 degrees toward 11-20 or 10-10 from a c-plane of said single crystal gallium nitride material.
36. The wafer of claim 26, finished by a process including at least one of lapping, polishing and CMP.
37. The wafer of claim 26, finished by a process including CMP.
38. The wafer of claim 26, wherein said one surface has a DDSDR of less than 50%.
39. The wafer of claim 26, wherein said one surface has a DDSDR of less than 25%.
40. The wafer of claim 26, wherein said one surface has a DDSDR of less than 10%.
41. The wafer of claim 26, having at least one epitaxial layer thereon.
42. The wafer of claim 41, wherein said at least one epitaxial layer comprises a heteroepitaxial layer.
43. The wafer of claim 41, wherein said at least one epitaxial layer comprises a homoepitaxial layer.
44. An electronic device article, including a wafer comprising large area single crystal gallium nitride material with uniformly low dislocation density on at least one surface thereof, and an electronic device structure fabricated on said wafer.

45. The electronic device article of claim 44, wherein the electronic device structure includes a laser diode.

46. The electronic device article of claim 44, wherein the electronic device structure includes a light-emitting diode.

47. The electronic device article of claim 44, wherein the electronic device structure includes a high electron mobility transistor.

48. The electronic device article of claim 44, wherein the electronic device structure comprises integrated circuitry.

49. The electronic device article of claim 44, wherein the electronic device structure includes an opto-electronic device.

50. Material according to claim 1, as grown under single crystal III-V nitride growth conditions slightly deviated from optimal single crystal III-V nitride growth conditions.

51. A wafer comprising material as claimed in claim 50.

52. A vapor phase growth process for forming a large area, uniformly low dislocation density single crystal III-V nitride material on a substrate, such process including (i) a first phase including one or more steps of growing the III-V nitride material on the substrate by a vapor phase growth technique under pitted growth conditions, and (ii) a second phase including one or more steps of growing the III-V nitride material by the vapor phase growth technique under pit-filling conditions effecting closure of pits and annihilation of defects on a growth surface of the III-V nitride material.

53. The process of claim 52, wherein the first phase growth forms pits over at least 50% of the growth surface of the III-V nitride material.
54. The process of claim 53, wherein the pit density on the growth surface is at least 100 pits/cm<sup>2</sup> of the growth surface at conclusion of the first phase growth.
55. The process of claim 52, wherein the second phase growth is continued until the growth surface has a pit density that does not exceed 10 pits/cm<sup>2</sup> of the growth surface.
56. The process of claim 52, wherein the second phase growth is continued until the growth surface comprises an essentially pit free surface.
57. The process of claim 52, wherein the second phase growth is continued until the growth surface has a pit density that does not exceed 1 pit/cm<sup>2</sup> of the growth surface.
58. The process of claim 52, wherein growth rate of the III-V nitride material on facets of the pits is greater than the growth rate in the c-axis direction in the second phase growth.
59. The process of claim 58, wherein the second phase growth is continued until the growth surface comprises an essentially pit free surface.
60. The process of claim 52, wherein the vapor phase growth technique comprises a technique selected from the group consisting of hydride vapor phase epitaxy (HVPE), metal-organic vapor phase epitaxy (MOVPE), metal-organic chloride method (MOC), molecular beam epitaxy (MBE), sublimation, sputtering, reactive sputtering, and reactive sublimation.
61. The process of claim 52, wherein the single crystal III-V nitride material is selected from the group consisting of AlN, InN, GaN, AlInN, AlInGaN, InGaN, and AlGaN.

62. The process of claim 52, wherein the single crystal III-V nitride material comprises GaN.
63. The process of claim 52, wherein the single crystal III-V nitride material comprises GaN and the vapor phase growth technique comprises HVPE.
64. The process of claim 52, wherein pit density on the growth surface is greater than 100 pits/cm<sup>2</sup> of the growth surface at conclusion of the first phase growth.
65. The process of claim 52, wherein pit density on the growth surface is greater than 1000 pits/cm<sup>2</sup> of the growth surface at conclusion of the first phase growth.
66. The process of claim 52, wherein growth rate of the III-V nitride material in a c-axis direction prior to the second phase growth is greater than growth rate in a facet direction on facets of pits formed on the growth surface.
67. The process of claim 52, wherein the first phase growth forms pits over at least 75% of the growth surface of the III-V nitride material
68. The process of claim 52, wherein the first phase growth forms pits over at least 90% of the growth surface of the III-V nitride material.
69. The process of claim 52, wherein pits formed on the growth surface in the first phase growth have an average size of greater than 25  $\mu\text{m}$  at the conclusion of the first phase growth.
70. The process of claim 52, wherein pits formed on the growth surface in the first phase growth have an average size of greater than 50  $\mu\text{m}$  at the conclusion of the first phase growth.

71. The process of claim 52, wherein pits formed on the growth surface in the first phase growth have an average size of greater than 100  $\mu\text{m}$  at the conclusion of the first phase growth.

72. The process of claim 52, wherein the first phase growth is conducted for sufficient time to grow the III-V nitride material to a thickness greater than 50  $\mu\text{m}$ .

73. The process of claim 52, wherein the first phase growth is conducted for sufficient time to grow the III-V nitride material to a thickness greater than 100  $\mu\text{m}$ .

74. The process of claim 52, wherein the first phase growth is conducted for sufficient time to grow the III-V nitride material to a thickness greater than 200  $\mu\text{m}$ .

75. The process of claim 52, wherein growth in the first phase growth is conducted at lower temperature than growth in the second phase growing step.

76. The process of claim 52, wherein transition from the first phase growth to the second phase growing step is abrupt.

77. The process of claim 52, wherein transition from the first phase growth to the second phase growth is gradual or multi-stepped.

78. The process of claim 52, wherein at least one of the first phase growth and the second phase growth includes more than one set of growth conditions.

79. The process of claim 52, wherein the first phase growth includes more than one set of growth conditions.



80. The process of claim 52, wherein the second phase growth includes a first stage of growth conditions producing filling of the pits produced in the first phase of growth conditions, followed by a second stage of growth conditions producing preferential growth on the c-plane of the III-V nitride material.

81. The process of claim 78, involving gradual transition between sets of growth conditions.

82. The process of claim 78, involving ramped transition between sets of growth conditions.

83. The process of claim 52, wherein the vapor phase growth technique comprises HVPE.

84. The process of claim 83, wherein the second phase growth comprises higher growth temperature and/or lower flow rate of ammonia than the first phase growth.

85. The process of claim 83, wherein second phase growth comprises higher growth temperature than the first phase growth.

86. The process of claim 83, wherein second phase growth comprises lower flow rate of ammonia than the first phase growth.

87. The process of claim 83, wherein second phase growth comprises lower flow ratio of ammonia to hydrogen chloride than the first phase growth.

88. The process of claim 52, wherein the second phase growth is conducted for sufficient time to grow the III-V nitride material to a thickness greater than 50  $\mu\text{m}$ .

89. The process of claim 52, wherein the second phase growth is conducted for sufficient time to grow the III-V nitride material to a thickness greater than 200  $\mu\text{m}$ .

90. The process of claim 52, wherein the second phase growth is conducted for sufficient time to grow the III-V nitride material to a thickness greater than 2 mm.

91. The process of claim 52, further comprising doping the III-V nitride material with a dopant species.

92. The process of claim 52, further comprising forming at least one wafer from the III-V nitride material after said second phase growth.

93. The process of claim 92, wherein the wafer includes a surface on axis with a c-plane of the III-V nitride material.

94. The process of claim 92, wherein the wafer includes an off-axis surface.

95. The process of claim 92, wherein the wafer includes a surface off-cut at an angle in a range of from about 0.2 to about 8 degrees toward 11-20 or 10-10 from a c-plane of said III-V nitride material.

96. The process of claim 52, further comprising doping the III-V nitride material to impart p-doped, n-doped or semi-insulatively doped character thereto.

97. The process of claim 52, further comprising doping the III-V nitride material with a dopant species including silicon and/or oxygen.

98. The process of claim 52, wherein the III-V nitride material has a large area of greater than 15 cm<sup>2</sup>.

99. The process of claim 52, wherein the III-V nitride material is grown to a thickness of at least 1 mm.

100. The process of claim 52, wherein the III-V nitride material at conclusion of the second phase growth has a dislocation density not exceeding  $1 \times 10^6 \text{ cm}^{-2}$ .

101. The process of claim 52, wherein the III-V nitride material at conclusion of the second phase growth has a dislocation density not exceeding  $5 \times 10^5 \text{ cm}^{-2}$ .

102. The process of claim 52, wherein the III-V nitride material at conclusion of the second phase growth has a DDSDR of less than 25%.

103. The process of claim 52, wherein the III-V nitride material at conclusion of the second phase growth has a DDSDR of less than 10%.

104. The process of claim 52, wherein the III-V nitride material is GaN, and the process is conducted under process conditions producing GaN having a large area of greater than  $15 \text{ cm}^2$ , a thickness of at least 1 mm, an ADD not exceeding  $1 \times 10^6 \text{ cm}^2$ , and a DDSDR of less than 25%.

105. The process of claim 52, wherein the III-V nitride material is grown on a heteroepitaxial substrate.

106. The process of claim 105, wherein the heteroepitaxial substrate comprises a material selected from the group consisting of sapphire, silicon carbide, gallium arsenide, silicon, lithium gallate, lithium aluminate, lithium aluminum gallate, zinc oxide, diamond, spinel, and magnesium oxide.

107. The process of claim 105, further comprising removing the heteroepitaxial substrate from the III-V nitride material.

108. The process of claim 107, wherein the heteroepitaxial substrate is removed from the III-V nitride material to produce a III-V nitride article of freestanding character.

109. The process of claim 108, wherein the heteroepitaxial substrate is removed from the III-V nitride material by a removal technique selected from the group consisting of: in situ etching of the substrate, at temperature within 100°C of growth temperature at conclusion of said growing steps; mechanical grinding of the substrate; and chemical grinding of the substrate.

110. The process of claim 52, wherein the III-V nitride material is grown on a homoepitaxial substrate.

111. The process of claim 52, wherein the growing steps are conducted under process conditions producing III-V nitride material in the form of an ingot.

112. The process of claim 111, wherein the ingot is grown to a thickness of at least 1 mm.

113. The process of claim 111, wherein the ingot is grown to a thickness greater than 5 mm.

114. The process of claim 111, wherein the ingot is grown to a thickness greater than 10 mm.

115. The process of claim 52, wherein the growing steps are conducted under process conditions producing III-V nitride material in the form of a single wafer body.

116. The process of claim 115, wherein the single wafer body is grown to a thickness of at least 50  $\mu\text{m}$ .

117. The process of claim 115, wherein the single wafer body is grown to a thickness greater than 200  $\mu\text{m}$ .

118. The process of claim 115, wherein the single wafer body is grown to a thickness greater than 500  $\mu\text{m}$ .

119. The process of claim 52, wherein the III-V nitride material is GaN, and the process is conducted under process conditions producing GaN having a diameter of from 2 to 8 inches.

120. A vapor phase growth process for forming a large area, uniformly low dislocation density single crystal III-V nitride material on a substrate, such process including (i) a first phase of growing the III-V nitride material on the substrate by a vapor phase growth technique under pitted growth conditions forming pits over at least 50% of the growth surface of the III-V nitride material wherein the pit density on the growth surface is at least 100/cm<sup>2</sup> of the growth surface at the end of the first phase, and (ii) a second phase of growing the III-V nitride material under pit-filling conditions that fill the pits to produce an essentially pit-free surface.